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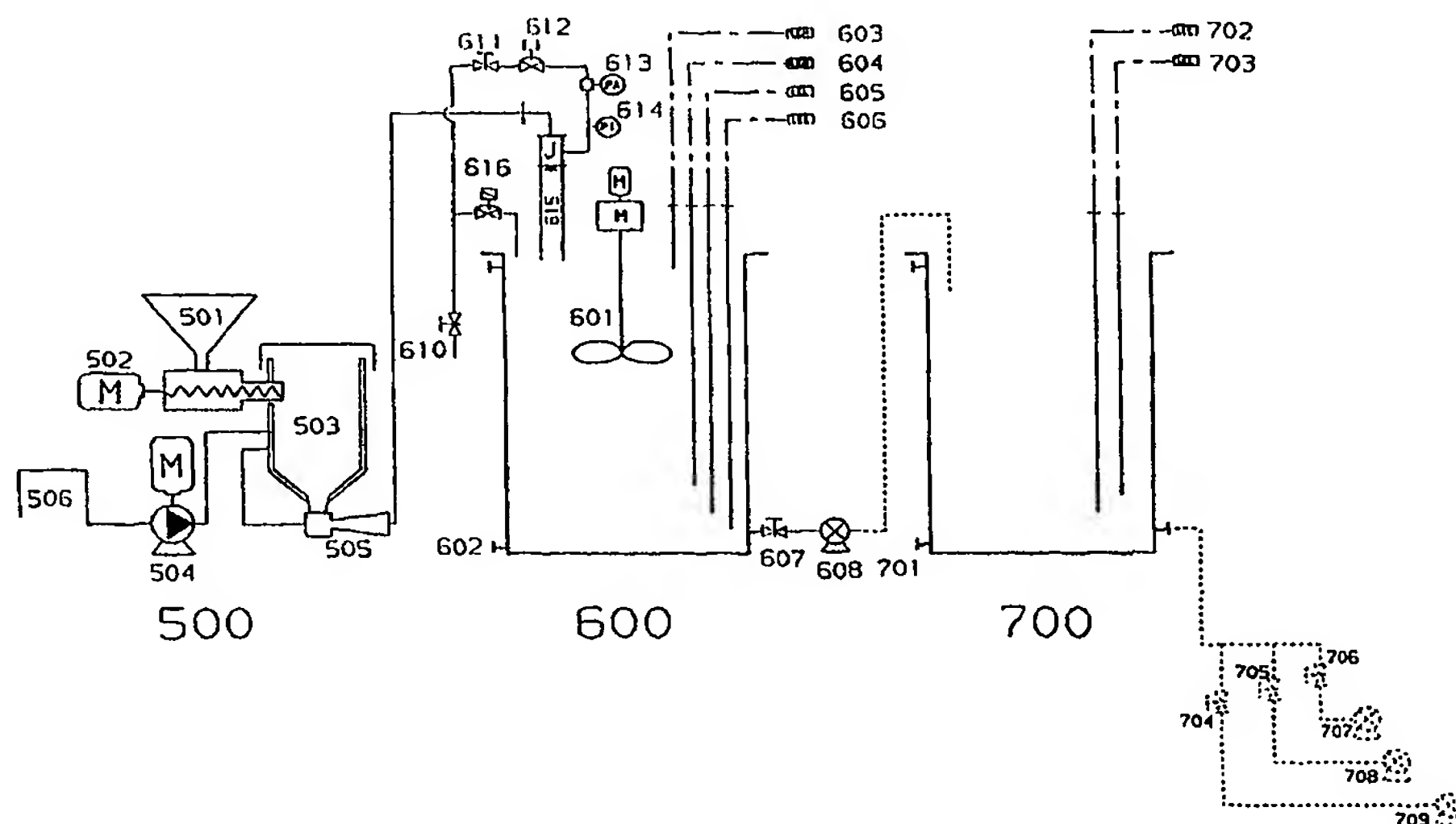
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(54) Title: DEWATERING OF AQUEOUS SUSPENSIONS WITH ANIONIC AND CATIONIC POLYMERS

**(57) Abstract**

A process for dewatering an aqueous suspension of solids which comprises adding to the suspension a flocculation aid, allowing the suspension to flocculate and dewatering the suspension, in which the flocculation aid is an aqueous composition comprising first and second water soluble ionic polymeric flocculants in which the first flocculant is in excess over the second flocculant and the first and second flocculants form particles of counterionic precipitate and substantially all of the proportion of the first flocculant which is not incorporated into the particles of counterionic precipitate is in solution, characterized in that the transfer and dosage conduits are constructed so that substantially none of the particles of counterionic precipitate are broken or dispersed after their formation and before their addition to the suspension.

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DEWATERING OF AQUEOUS SUSPENSIONS WITH ANIONIC AND CATIONIC POLYMERS

The present invention relates to processes for flocculation and subsequent dewatering (including thickening) of aqueous suspensions. In particular it relates to such processes in which flocculation aids are activated and dosed into the suspension in a defined manner which ensures optimum performance of the flocculation aid.

It is well known to dewater aqueous suspensions of solids, both inorganic (or mineral) and organic, in water by the addition of a flocculation aid, allowing the solids to flocculate and dewatering the suspension.

In our International Publication WO97/06111 we describe one such method in which an aqueous mineral suspension having high solids content (at least 150 g/l) is dewatered under pressure after flocculation using a composition made by blending one part by weight cationic polymeric flocculant, 2 to 20 parts by weight anionic polymeric flocculant and water. In the composition counterionic precipitation occurs and all the anionic polymer which is not precipitated by the cationic polymer is in solution. In preferred processes the counterionic precipitate takes the form of particles in which anionic polymeric flocculant forms a coacervate around the cationic polymeric flocculant. In that publication we explain that activation of the polymers should be gentle and without application of sufficient shear to disperse any counterionic precipitate which is formed.

We also describe in our co-pending British patent application (not yet published) number 9801524.1 processes for thickening of aqueous suspensions in which similar aqueous compositions are used as the flocculation aid. In our co-pending UK application number 9807047.7 filed 01 April 1998, reference PRL04083GB, we describe dewatering of organic suspensions in which aqueous compositions of this type can be used. In UK application 9807047.7 aqueous compositions containing counterionic precipitate particles, in which cationic flocculant is in

excess can also be used as the flocculation aid.

We have now found a particularly advantageous method of ensuring gentle activation so that the particles of counterionic precipitate are not broken or dispersed before dosing into the suspension.

According to a first aspect of the invention we provide a process for dewatering an aqueous suspension of solids which comprises adding to the suspension a flocculation aid, allowing the suspension to flocculate and dewatering the suspension, in which the flocculation aid is an aqueous composition comprising first and second water soluble ionic polymeric flocculants in which the first flocculant is in excess over the second flocculant and the first and second flocculants form particles of counterionic precipitate and substantially all of the proportion of the first flocculant which is not incorporated into the particles of counterionic precipitate is in solution,

and in the process the aqueous composition is added to the suspension by

(a) adding to water the first and second flocculants in powder form

(b) mixing the powders into the water in a mixing vessel so as to form an aqueous composition containing particles of counterionic precipitate,

(c) passing the aqueous composition from the mixing vessel into a transfer conduit

(d) optionally passing the aqueous composition from the transfer conduit into a storage vessel,

(e) passing the aqueous composition from the storage vessel, if used, or from the transfer conduit, into a dosage conduit, and

(f) passing the aqueous composition from the dosage conduit into the suspension,

characterised in that the transfer and dosage conduits are constructed so that substantially none of the particles of counterionic precipitate are broken or dispersed after their formation and before their addition

to the suspension.

We have found surprisingly that the construction of the transfer and dosage conduits can be highly influential in ensuring that the particles of counterionic precipitate are maintained from the point of their formation to the point of its addition to the suspension. We find this particularly surprising given that in other stages of the make-up and dosing process the aqueous composition is often subjected to levels of shear which are conventionally used for the make-up of conventional aqueous polymer solutions which do not contain counterionic precipitate.

In the process the two counterionic flocculants in the form of powder, a reverse phase emulsion or a reverse phase dispersion are added to water in a mixing vessel. The aqueous composition containing counterionic precipitate and dissolved excess first polymeric flocculant is formed in the mixing vessel. From the mixing vessel the aqueous composition is passed to the transfer conduit. This is often achieved with the use of a transfer pump. Alternatively, a transfer valve or other transfer means operating under gravity can be used. The aqueous composition is passed along the transfer conduit. Optionally and preferably the aqueous composition may then be passed to a storage vessel from the transfer conduit. If, as is preferred, a storage vessel is used, the aqueous composition is transferred from the storage vessel to a dosage conduit, often with the use of a dosage pump, and along the dosage conduit to the aqueous suspension. If a storage vessel is not provided the aqueous composition is transferred directly from the transfer conduit to the dosage conduit and into the suspension. In this case the transfer and dosage conduits thus effectively form two parts of a single conduit.

In the invention the transfer and dosage conduits are constructed such that the particles of counterionic precipitate formed during the mixing stage are not substantially broken up or dispersed. In particular, it is

preferred that at least 80% by weight of the particles, more preferably at least 90 or 95% by weight of the particles, remain intact after passage of the aqueous composition through the transfer and dosage conduits, so
5 that they are present in the aqueous composition which is added to the suspension.

The conduits may be any suitable type of conduit, for instance open channels, or, preferably, pipes. Pipes can be of any suitable cross-section but are preferably
10 substantially cylindrical.

The required stability of the particles of counterionic precipitate can be obtained in the invention in various ways. A preferred method is by choice of the conformation of the conduits.

15 In conventional processes for dewatering aqueous suspensions, conduits are generally provided so as to provide the most convenient pathway between two points. It is not usually possible to arrange a conduit between two points in a straight line, and bends are usually required
20 in the conduits. Thus conduits present for use in dewatering processes are those already present and configured for transfer of conventional solutions. It is conventional practice in dewatering processes simply to provide any bends in the form of sharp corners joining two
25 essentially straight sections of conduit at angles often around 90°. The conventional pathway followed by a conduit between a mixing vessel and a storage vessel or dosage point may have 3 or 4 such bends.

We have found that the stability of the particles of
30 counterionic precipitate can in some cases be improved by the provision of long radius bends or very long radius bends, i.e. curved rather than sharp bends. For instance, in the invention the transfer and dosage conduits preferably follow a pathway which contains not more than
35 two sharp bends and especially not more than two sharp bends at an angle below 120°, in particular 100° or less.

Preferably all bends in the pathway followed by the transfer and dosage conduits are long radius bends or very long radius bends.

5 Bends having a centre to face (i.e. edge) dimension of at least 1.5 times the conduit diameter, for instance at least 2 or 3 times and up to 5 times the conduit diameter, are particularly useful.

10 In particular we find that the provision of conduits which follow bends having a radius of curvature higher than has been used previously for such processes is particularly advantageous. Radius of curvature at least 1.5 times, preferably at least twice the diameter of the conduit, and even at least 3 or 5 times the diameter, can be used.

15 The required bends having larger radius of curvature than the standard 90° sharp bends can be constructed in various ways.

20 The conduit, for instance pipework, having radius of curvature higher than conventional, may be formed from rigid material. In this case the pipe sections at the bend have the required radius of curvature. These are often referred to as long radius bends or very long radius bends.

25 Alternatively the pipework can be formed from flexible material. In this case the pipework can be bent into the required curve at the bend. Portions of the flexible pipework may be surrounded by rigid casing which is usually substantially concentric with the flexible pipework. At the points where a bend is required there is no casing so that the flexible inner pipework can be curved around the required bend.

30 Alternatively or additionally it has been found that control of the bore diameter of conduits such as pipework can influence the stability of the particles of counterionic precipitate. For instance, the bore diameter may be 120% or more of the diameter of the piping which
35 would normally be used as a conduit for flocculation aids which do not contain particles of counterionic precipitate. For instance a bore size of 130 to 200% of this

conventional diameter can be used. Standard pipes often have diameter 20 to 50 mm, and in the invention diameters 40 to 75 mm and even up to 100 mm can be used.

5 The internal surface of the conduit, across which the aqueous composition containing the particles of counterionic precipitate passes during transfer from mixing to dosage, can also be influential. In particular surfaces which are smooth and have low coefficient of friction are preferred. Conventional pipework is often formed from mild
10 steel. In the invention on the other hand the internal surface of the conduit may be formed from a corrosion resistant and/or rust resistant material such as galvanised metal. Alternatively it may be formed of a plastic material.

15 The length of the pathway followed by the conduit between the mixing vessel and storage vessel, if used, is preferably 1 to 10m, more preferably 1 to 5m.

The processes of the invention are especially advantageous when the aqueous composition contains
20 counterionic precipitate particles of size in the aqueous composition 90 wt.% above 100 microns, preferably above 200 microns, for instance above 300 microns and up to 1mm.

In particular we find that control of the construction of the transfer and dosage conduits is
25 advantageous when the counterionic precipitate particles are in the form of a coacervate of a polymeric flocculant of one ionic type around a polymeric flocculant of the opposite ionic type. Usually the core flocculant is the second flocculant and the coacervating flocculant is the
30 first flocculant, which is in excess. The flocculants are counterionic, i.e. one is cationic and one is anionic. Either may be the first flocculant, which is in excess. Preferably the first flocculant is anionic.

Preferred processes use the aqueous composition
35 described in our International Publication WO97/06111 in which the anionic polymeric flocculant is in excess and is present in an amount of from 2 to 20 times by weight the

amount of cationic polymeric flocculant and in which the anionic polymeric flocculant forms a coacervate around a core of cationic polymeric flocculant to form the particles of counterionic precipitate.

5 The two polymer types are preferably added to water in step (a) as a preformed powder blend, and this blend mixed into water in step (b).

10 The anionic polymeric flocculant may be formed from water soluble ethylenically unsaturated anionic monomer or monomer blend. Generally at least 3 wt.%, often at least 5, 10 or 15 wt.% but generally not more than 50 or 60 wt.% of the monomers are anionic with any other monomers being nonionic. In this specification we include within the definition of "anionic flocculant" polymers which are often
15 referred to as "substantially nonionic" and contain for instance 1 or 2 wt.% anionic monomer. These monomers may be present due to hydrolysis of non-ionic monomer such as acrylamide. These polymers are encompassed within the term "anionic flocculant" provided that they form a counterionic
20 precipitate with the cationic flocculant which is used.

 The anionic monomers which can be used can be any of those described in our publication WO97/06111.

 The anionic polymeric flocculant is preferably a high molecular weight bridging flocculant as described in
25 WO97/06111.

 The cationic polymeric flocculant may also be any of those discussed in WO97/06111. We also include within the definition of "cationic flocculant" polymers which are sometimes described as "substantially nonionic" and have
30 low content of cationic monomer, for instance 1 or 2 wt.%, provided that they form a counterionic precipitate with the anionic flocculant which is used.

 Preferably both the anionic and cationic flocculants are of high molecular weight, that is the anionic polymeric
35 material has IV preferably at least about 5 dl/g and the cationic polymer has intrinsic viscosity preferably at least 4dl/g.

In this specification intrinsic viscosity is measured by suspended level viscometer in buffered pH 7 1M NaCl at 25°C.

5 The total polymer concentration in the aqueous composition can be any of the concentrations discussed in the applications WO97/06111, GB 9801524.1 and GB 9807047.7. Preferred polymer concentration is from 0.05 to 1%, in particular 0.1 to 0.5%, for instance around 0.3%.

10 The polymer powders can be produced in any conventional manner. Gel polymerisation followed by comminution, and reverse phase bead polymerisation, optionally followed by comminution, are preferred.

15 In this specification we include within the term "dewatering", processes of dewatering to form a cake, such as dewatering under pressure, for instance by belt pressing, filter pressing, vacuum filtration and centrifugation etc, and processes often described as thickening processes.

20 The overall process may be a dewatering process as described in WO97/06111.

It may also be a process as described in our co-pending application no. 9801524.1 of thickening an aqueous suspension of mineral solids by sedimentation by adding to the suspension water-soluble anionic bridging polymeric
25 flocculant having intrinsic viscosity at least 5 dl/g and water soluble cationic polymeric flocculant, allowing the solids to settle under gravity so as to form a lower underflow fraction containing a concentration of solids greater than that of the suspension and an upper overflow
30 fraction containing a concentration of solids lower than that of the suspension and removing the underflow fraction, in which the anionic and cationic flocculants are added to the suspension as an aqueous composition formed by blending one part by weight of the cationic polymeric flocculant
35 with 2 to 20 parts by weight of the anionic polymeric flocculant and sufficient water to give a total polymer concentration of below 5 wt.% and under conditions whereby

counterionic precipitation can occur and in the aqueous composition substantially all of the anionic polymeric flocculant which is not precipitated by the cationic polymeric flocculant is in solution.

5 Any of the features of the processes described in these two applications can be applied in the process of the present invention.

The invention can also be used in a process as described in our co-pending patent application number GB
10 9807047.7 filed 01 April 1998, which describes a process of dewatering a suspension of organic suspended solids comprising adding to the suspension an aqueous composition comprising a first water-soluble ionic polymeric flocculant and a second water-soluble ionic polymeric flocculant in
15 which the first flocculant is in excess over the second flocculant and the first and second flocculants are counterionic and in the aqueous composition at least a portion of the first flocculant and at least a portion of the second flocculant together form particles of
20 counterionic precipitate, allowing the suspended solids to flocculate and dewatering the flocculated suspension, in which the particles of counterionic precipitate are of size at least 90 wt.% above 100 microns in the aqueous composition and substantially all of the portion of the
25 first flocculant which is not contained in the particles of counterionic precipitate is in solution.

The process of the present invention may be applied to any of the processes described in that application.

We find it particularly surprising that the design of
30 the conduit in the process is so effective in maintaining stability of the particles of counterionic precipitate in processes in which mixing and pumping are under conditions of fairly rapid rotation. In particular the mixing vessel may be operated at the level of shear provided by a
35 impeller shaft of length 1 to 2 metres, for instance about 1.8 metres, and diameter 20 to 60mm, for instance about 40mm having impeller blades (e.g. 3 blades of diameter 300

to 400mm, e.g. 345mm) and rotating at a speed of from 50 to 300 rpm, for instance 200 to 290 rpm, in a mixing vessel of volume 3 to 20m³, for instance around 5m³. Other agitation methods which give equivalent levels of shear, such as air
5 agitation and use of a reciprocating impeller, may also be used. It is surprising that particles of counterionic precipitate which have been produced in a process in which the aqueous composition is subjected to shear as discussed above at rotation speeds of up to 290 rpm without breakage
10 or dispersal can be affected to such a large extent by the construction of the conduit. Mixing vessels of volume below 3m³ can also be used, but less preferably.

The aqueous composition is transferred from the mixing vessel to the transfer conduit using any suitable
15 means, for instance a transfer valve or a transfer pump. If the mixing vessel is elevated with respect to the dosage point or the storage vessel (if used), a valve which operates under gravity can be used. A transfer pump is preferred. Preferred pumps are mono progressive cavity
20 pumps which can operate at speeds up to 450 rpm. Speed is normally at least 50 rpm and preferred speeds are about 400 rpm.

If a storage vessel is used this can be of conventional type. However, we do find that it is
25 advantageous to use a storage vessel provided with agitation means in processes in which the aqueous composition contains particles of counterionic precipitate.

Thus according to a second aspect of the invention we provide a process for dewatering an aqueous suspension of
30 solids comprising adding to the suspension a flocculation aid, allowing the suspension to flocculate and dewatering the suspension, in which the flocculation aid is an aqueous composition comprising first and second water-soluble ionic polymeric flocculants in which the first flocculant is in
35 excess over the second flocculant and the first and second flocculants form particles of counterionic precipitate and substantially all of the portion of the first flocculant

which is not incorporated into the particles of counterionic precipitate is in solution,

and in the process the aqueous composition is added to the suspension by

5 (a) adding to water the first and second flocculants in powder form,

(b) mixing the powders into water in a mixing vessel so as to form an aqueous composition containing particles of counterionic precipitate,

10 (c) passing the aqueous composition from the mixing vessel into a transfer conduit,

(d) passing the aqueous composition from the transfer conduit into a storage vessel,

15 (e) passing the aqueous composition from the storage vessel into a dosage conduit, and

(f) passing the aqueous composition from the dosage conduit to the suspension,

characterised in that the aqueous composition is subjected to low shear agitation in the storage vessel by
20 air agitation or by intermittent mechanical agitation.

It is important in this aspect of the invention that the aqueous composition is not subjected to standard high shear agitation. This aspect of the invention is particularly useful for aqueous compositions in which the
25 counterionic precipitate particles have a tendency to settle if not agitated and in which the aqueous composition may need to be stored for significant periods of time, for instance 1 to 2 days or even up to two weeks.

When air agitation is used the rate of air flow can
30 be from 2 to 4 litres per second, for instance about 3 litres per second. These ranges are suitable for storage vessels of volume about 5 m³, for instance 1 to 10 m³. For storage vessels of volume from 10 or 15 to 25 m³ the air flow rate can be from around 10 to 20 litres per second,
35 for instance around 15 litres per second.

Low shear intermittent mechanical mixing can be used. A rotating impeller can be used for this purpose at a

rotation rate of up to 290 rpm. Intermittent mixing should be used, for instance for a period of 1 to 10 minutes every 1 to 6 hours, e.g. 2 minutes in every 4 hours.

Any of the process features discussed in connection with the first aspect of the invention may also be applied to this second aspect of the invention.

According to a third aspect of the invention we provide a process of modifying an apparatus to render it suitable for use in the process of the first aspect of the invention by modifying the transfer or dosage conduit so that at least one, and preferably all, bends in the conduit are modified by increasing their radius of curvature.

According to a fourth aspect of the invention we modify an apparatus to render it suitable in the process of the first aspect of invention by increasing the bore size of a pipe which is a transfer or dosage conduit by at least 20%, preferably at least 50%.

According to a fifth aspect of the invention we modify an apparatus to render it suitable for use in the process of the first aspect of the invention by reducing the coefficient of friction of the inner surface of the transfer and dosage conduits.

A process according to the invention is illustrated schematically in the attached Figure 1. Powdered anionic and cationic polymeric flocculant are held in the powder hopper 501 and passed using a screw feeder 502 into a venturi inlet 503. The ratio of anionic to cationic flocculants is approximately 9:1 by weight. By means of a blower 504 the polymer powder is passed from the venturi eductor to the "Jet Wet" head. The "Jet Wet" device is a commercially available device for dissolving polymer powder into water and available from Ciba Specialty Chemicals Water Treatments Limited. The polymer powder is injected from the Jet Wet head into the mixer 600 which contains water, into which the polymer powder is mixed using the mixer 601. In the mixing tank 600 the aqueous composition containing dissolved anionic flocculant and counterionic

precipitate particles is formed. The mixing tank contains a shaft of diameter 40mm and length 1800mm having 3 impeller blades and two sets of blades per shaft. The shaft rotates at a rate of up to 290 rpm.

5 From the mixing tank the aqueous composition passes into the transfer pump 608. This is a mono progressive cavity pump operating at a speed of approximately 400 rpm.

10 From the transfer pump the aqueous composition is passed into the transfer conduit 617, shown schematically as a dotted line into the storage tank 700. In this embodiment the storage tank is not agitated but in other embodiments it can be air agitated at a rate of approximately 3 litres per second. From the storage tank the aqueous composition passes into the dosage conduit 710
15 and to the dosing pumps 707, 708 and 709 from where it is dosed into the aqueous suspension.

 In Figure 1 the following key applies:-

- 500 Screw Feeder & Powder Conveyor
- 501 Powder Hopper
- 20 502 Screw Feeder
- 503 Venturi Inlet
- 504 Blower
- 505 Venturi Eductor
- 506 Blower Inlet
- 25 600 Mixing Tank
- 601 Mixer
- 602 Drain
- 603 High Level Electrode
- 604 Intermediate level Electrode
- 30 605 Low Level Electrode
- 606 Earth Electrode
- 607 Transfer Pump Isolating Valve
- 608 Transfer Pump
- 610 Water Inlet Valve
- 35 611 Jet Wet Regulating Valve
- 612 Jet Wet Solenoid Valve
- 613 Low Water Pressure Alarm Switch

- 614 Jet Wet Pressure Gauge
- 615 Jet Wet Head
- 616 Rapid Fill Solenoid Valve
- 700 Storage Tank
- 5 701 Drain
- 702 Earth Electrode
- 703 Storage Tank Level Electrode
- 704-706 Dosing Pump Isolating Valves
- 707-709 Dosing Pumps

10

Figure 2 shows the shape of a conventional elbow bend and Figure 3 shows the shape of the bends used in the transfer and dosage conduits in the invention.

Claims

1. A process for dewatering an aqueous suspension of solids which comprises adding to the suspension a flocculation aid, allowing the suspension to flocculate and dewatering the suspension, in which the flocculation aid is an aqueous composition comprising first and second water soluble ionic polymeric flocculants in which the first flocculant is in excess over the second flocculant and the first and second flocculants form particles of counterionic precipitate and substantially all of the proportion of the first flocculant which is not incorporated into the particles of counterionic precipitate is in solution,

and in the process the aqueous composition is added to the suspension by

(a) adding to water the first and second flocculants in powder form

(b) mixing the powders into the water in a mixing vessel so as to form an aqueous composition containing particles of counterionic precipitate,

(c) passing the aqueous composition from the mixing vessel into a transfer conduit

(d) optionally passing the aqueous composition from the transfer conduit into a storage vessel,

(e) passing the aqueous composition from the storage vessel, if used, or from the transfer conduit, into a dosage conduit, and

(f) passing the aqueous composition from the dosage conduit into the suspension,

characterised in that the transfer and dosage conduits are constructed so that substantially none of the particles of counterionic precipitate are broken or dispersed after their formation and before their addition to the suspension.

2. A process according to claim 1 in which the particles of counterionic precipitate are maintained from the point of their formation to the point of its addition to the suspension.

3. A process according to claim 1 or claim 2 in which the aqueous composition is formed by mixing into water the cationic and anionic polymers in powder form, reverse phase emulsion form or reverse phase dispersion form.
- 5 4. A process according to any of claims 1 to 3 in which the transfer and dosage conduits follow a pathway which contains not more than two sharp bends and especially not more than two sharp bends at an angle below 120°, in particular 100° or less.
- 10 5. A process according to any of claims 1 to 4 in which provision of conduits which follow bends having a radius of curvature at least 1.5 times, preferably at least twice the diameter of the conduit.
- 15 6. A process according to any of claims 1 to 5 in which the internal surface of the conduit is formed from a corrosion resistant and/or rust resistant material such as galvanised metal or a plastic material.
- 20 7. A process according to any of claims 1 to 6 in which the anionic polymeric material has IV at least about 5 dl/g and the cationic polymer has intrinsic viscosity at least 4dl/g.
8. A process according to any of claims 1 to 7 in which the dewatering is by belt pressing, filter pressing, vacuum filtration and centrifugation.
- 25 9. A process according to any of claims 1 to 8 in which the aqueous composition is made in a mixing vessel comprising a impeller shaft having impeller blades which rotates at a speed of from 50 to 300 rpm.
- 30 10. A process according to any of claims 1 to 9 in which the aqueous composition is made in a mixing vessel of volume 3 to 20m³, preferably around 5m³.
11. A process according to any of claims 1 to 10 in which the aqueous composition is transferred from the mixing vessel to the transfer conduit using a transfer valve or a transfer pump.
- 35 12. A process according to claim 11 in which the aqueous composition is transferred from the mixing vessel to the

transfer conduit using a mono progressive cavity pumps.

13. A process according to claim 11 or claim 12 in which the aqueous composition is transferred from the mixing vessel to the transfer conduit using a transfer pump which
5 operates at between 50 and 450 rpm, preferably about 400 rpm.

14. A process for dewatering an aqueous suspension of solids comprising adding to the suspension a flocculation aid, allowing the suspension to flocculate and dewatering
10 the suspension, in which the flocculation aid is an aqueous composition comprising first and second water-soluble ionic polymeric flocculants in which the first flocculant is in excess over the second flocculant and the first and second flocculants form particles of counterionic precipitate and
15 substantially all of the portion of the first flocculant which is not incorporated into the particles of counterionic precipitate is in solution,

and in the process the aqueous composition is added to the suspension by

20 (a) adding to water the first and second flocculants in powder form,

(b) mixing the powders into water in a mixing vessel so as to form an aqueous composition containing particles of counterionic precipitate,

25 (c) passing the aqueous composition from the mixing vessel into a transfer conduit,

(d) passing the aqueous composition from the transfer conduit into a storage vessel,

30 (e) passing the aqueous composition from the storage vessel into a dosage conduit, and

(f) passing the aqueous composition from the dosage conduit to the suspension,

characterised in that the aqueous composition is subjected to low shear agitation in the storage vessel by
35 air agitation or by intermittent mechanical agitation.

15. A process according to claim 14 in which the aqueous composition is stored in the storage vessel for at least

1 day.

16. A process according to claim 14 or claim 15 in which the intermittent mixing is for a period of 1 to 10 minutes every 1 to 6 hours.

5 17. A process of modifying an apparatus to render it suitable for use in the process of claim 1 by modifying the transfer or dosage conduit so that at least one, and preferably all, bends in the conduit are modified by increasing their radius of curvature.

10 18. A process of modifying an apparatus to render it suitable in the process of claim 1 by increasing the bore size of a pipe which is a transfer or dosage conduit by at least 20%, preferably at least 50%.

15 19. A process of modifying an apparatus to render it suitable for use in the process of claim 1 by reducing the coefficient of friction of the inner surface of the transfer and dosage conduits.

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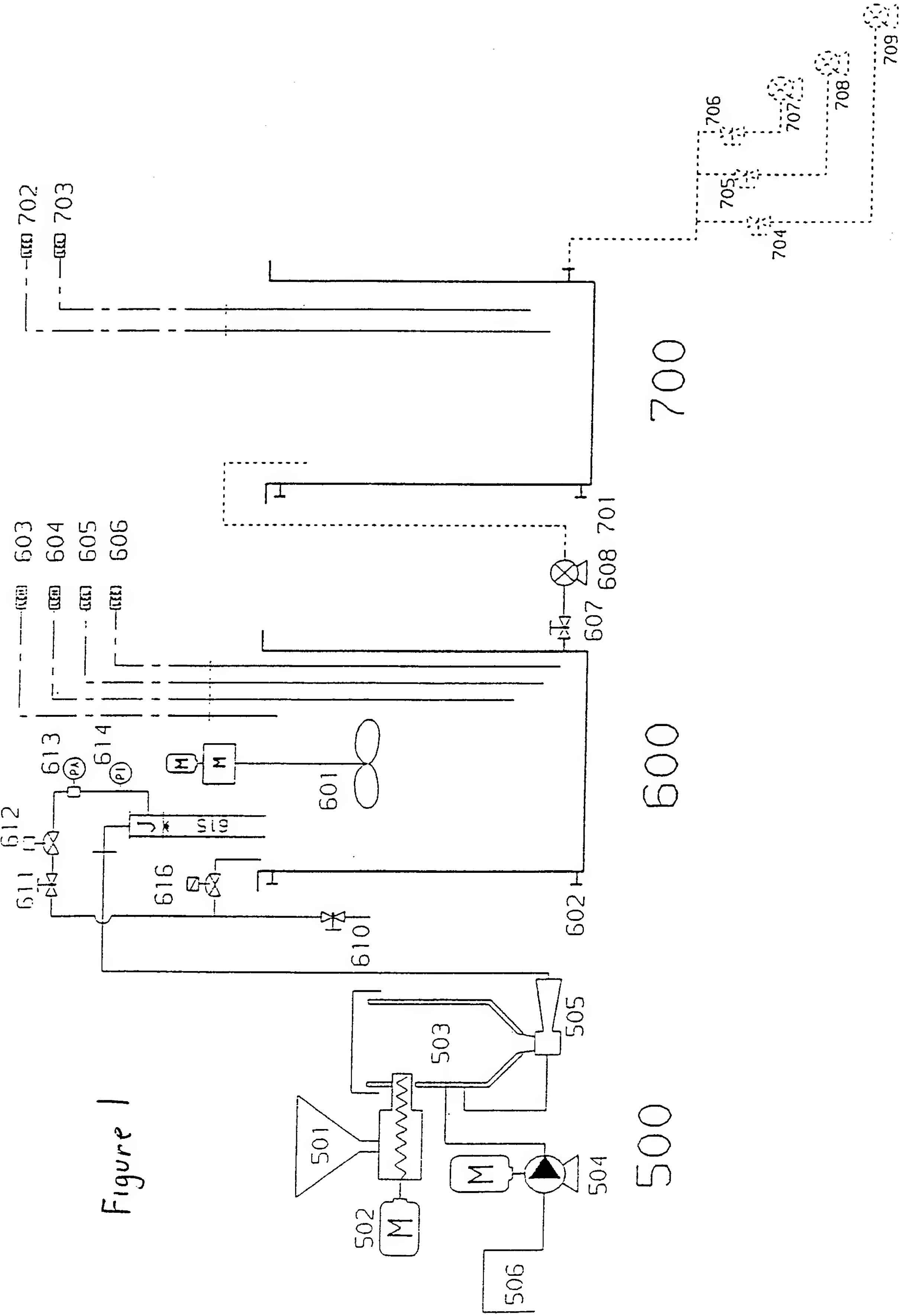


Figure 2

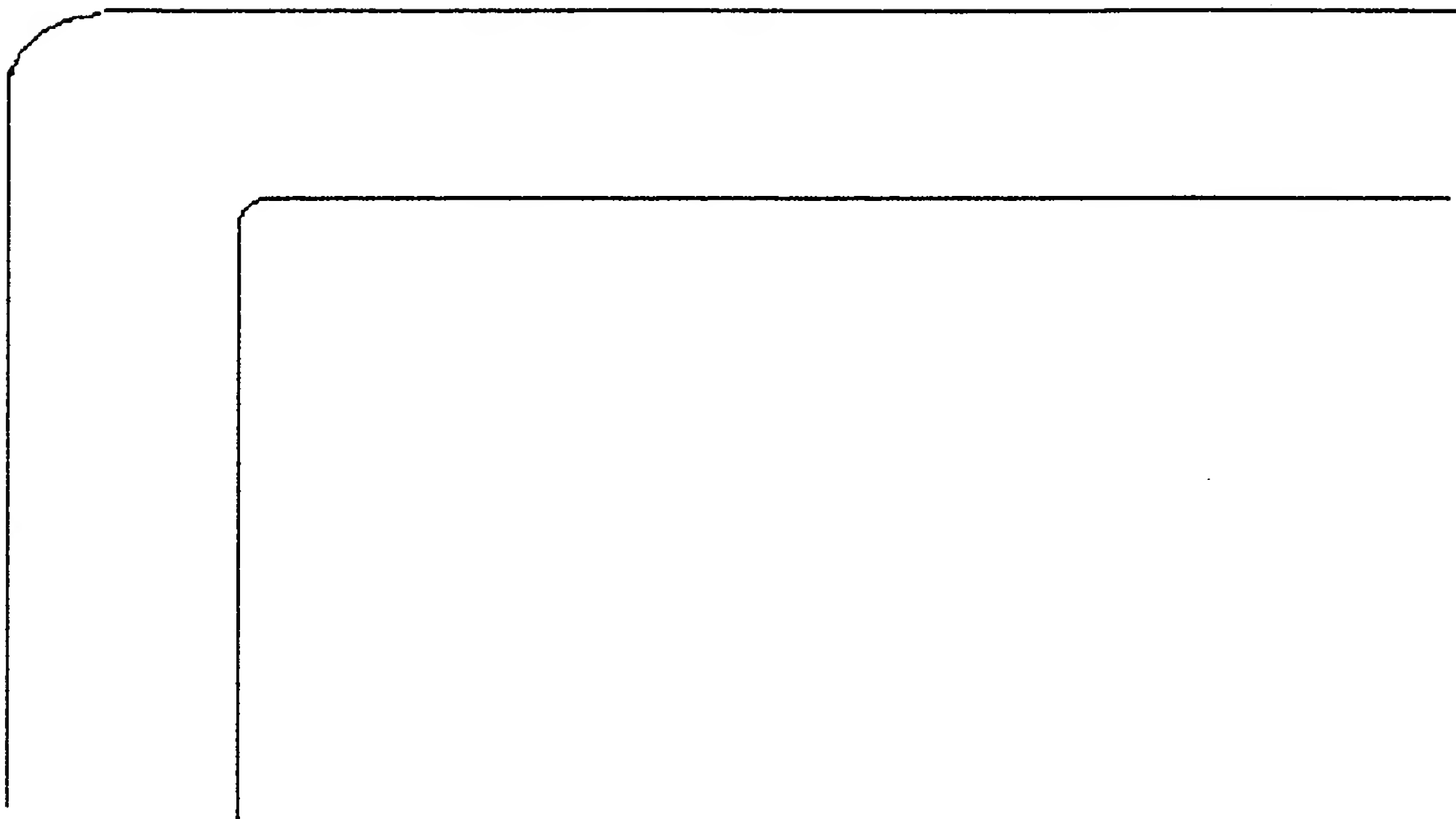
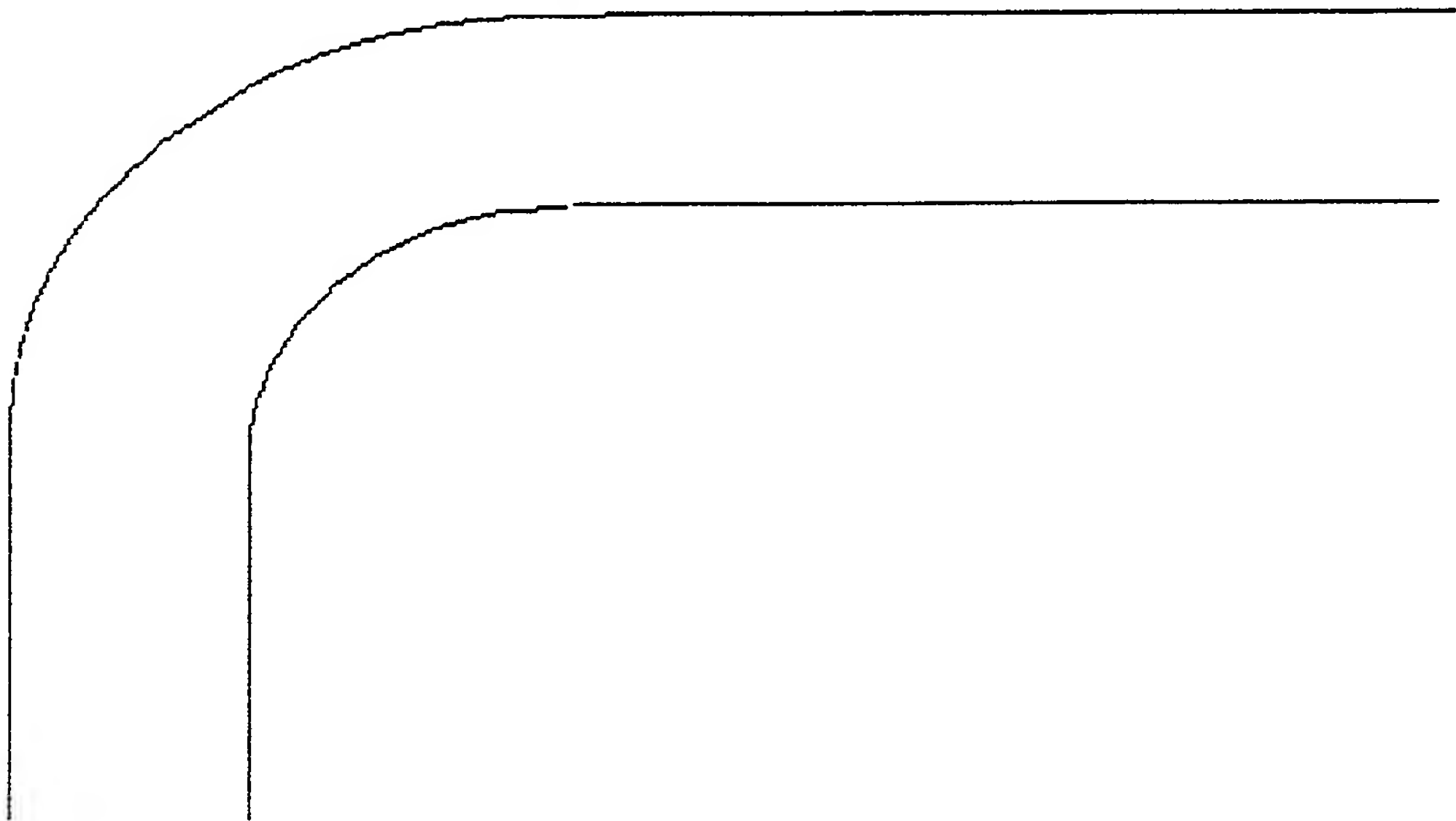


Figure 3



INTERNATIONAL SEARCH REPORT

In ternational Application No

PCT/GB 99/00990

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C02F1/56 C02F11/14 B01D21/01

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C02F B01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4 759 856 A (FLESHER PETER ET AL) 26 July 1988 (1988-07-26) column 7, line 23-61; claims 1,5 ---	1-3,7,8, 10,14
A	EP 0 510 517 A (STRANCO INC) 28 October 1992 (1992-10-28) the whole document ---	1,4-6,9, 11-14
A	WO 97 06111 A (ALLIED COLLOIDS LTD ;ALLEN ANTHONY PETER (GB); MCCOLL PHILIP (GB);) 20 February 1997 (1997-02-20) the whole document ---	1-3,7,8, 10,14
A	US 4 720 346 A (FLESHER PETER ET AL) 19 January 1988 (1988-01-19) column 7, line 24-54 column 11, line 28-42 ---	1-3,7,8, 14-16
-/--		

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

16 July 1999

Date of mailing of the international search report

04/08/1999

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 99/00990

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4 043 955 A (PASTER MARK D) 23 August 1977 (1977-08-23) the whole document ---	1,4-6,9, 11-13, 17-19
A	DE 43 11 837 A (MOOS SIMON MASKIN) 13 October 1994 (1994-10-13) figure 1 -----	1,4-6, 10,11, 13,14

INTERNATIONAL SEARCH REPORT

Information on patent family members

In ternational Application No

PCT/GB 99/00990

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4759856 A	26-07-1988	CA 1255016 A	30-05-1989
		EP 0160527 A	06-11-1985
		JP 1915733 C	23-03-1995
		JP 6038882 B	25-05-1994
		JP 61018408 A	27-01-1986
		US 4705640 A	10-11-1987
		AU 5658386 A	06-11-1986
		AU 594671 B	15-03-1990
		AU 5658486 A	06-11-1986
		AU 581980 B	09-03-1989
		AU 5658586 A	06-11-1986
		CA 1277449 A	04-12-1990
		CA 1284933 A	18-06-1991
		CA 1277785 A	11-12-1990
		DK 188886 A,B,	26-10-1986
		DK 188986 A	26-10-1986
		DK 189086 A,B,	26-10-1986
		EP 0207592 A	07-01-1987
		EP 0201237 A	12-11-1986
		EP 0202780 A	26-11-1986
		FI 861722 A	26-10-1986
		FI 861723 A,B,	26-10-1986
		FI 861724 A	26-10-1986
		JP 2087416 C	02-09-1996
		JP 8000163 B	10-01-1996
		JP 61293509 A	24-12-1986
		JP 1986445 C	08-11-1995
		JP 7018871 B	06-03-1995
		JP 61275653 A	05-12-1986
		JP 8000164 B	10-01-1996
		JP 61293510 A	24-12-1986
		US 4943378 A	24-07-1990
		US 4720346 A	19-01-1988
		US 4820645 A	11-04-1989
EP 0510517 A	28-10-1992	AU 643171 B	04-11-1993
		AU 1507292 A	29-10-1992
		CA 2067020 A	26-10-1992
		US 5316031 A	31-05-1994
		ZA 9202954 A	25-10-1993
WO 9706111 A	20-02-1997	AU 691124 B	07-05-1998
		AU 6665496 A	05-03-1997
		AU 703657 B	01-04-1999
		AU 7878898 A	01-10-1998
		CA 2223855 A	20-02-1997
		EP 0861214 A	02-09-1998
		PL 324895 A	22-06-1998
US 4720346 A	19-01-1988	AU 5658386 A	06-11-1986
		AU 594671 B	15-03-1990
		AU 5658486 A	06-11-1986
		AU 581980 B	09-03-1989
		AU 5658586 A	06-11-1986
		CA 1277449 A	04-12-1990
		CA 1284933 A	18-06-1991
		CA 1277785 A	11-12-1990
		DK 188886 A,B,	26-10-1986

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 99/00990

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4720346 A		DK 188986 A	26-10-1986
		DK 189086 A,B,	26-10-1986
		EP 0207592 A	07-01-1987
		EP 0201237 A	12-11-1986
		EP 0202780 A	26-11-1986
		FI 861722 A	26-10-1986
		FI 861723 A,B,	26-10-1986
		FI 861724 A	26-10-1986
		JP 2087416 C	02-09-1996
		JP 8000163 B	10-01-1996
		JP 61293509 A	24-12-1986
		JP 1986445 C	08-11-1995
		JP 7018871 B	06-03-1995
		JP 61275653 A	05-12-1986
		JP 8000164 B	10-01-1996
		JP 61293510 A	24-12-1986
		US 4943378 A	24-07-1990
		US 4820645 A	11-04-1989
		US 4759856 A	26-07-1988
US 4043955 A	23-08-1977	BE 838707 A	19-08-1976
		CA 1042127 A	07-11-1978
		DE 2606715 A	02-09-1976
		FR 2301552 A	17-09-1976
		GB 1521805 A	16-08-1978
		JP 1304950 C	28-02-1986
		JP 51107340 A	22-09-1976
		JP 60025451 B	18-06-1985
DE 4311837 A	13-10-1994	AT 159921 T	15-11-1997
		CA 2160007 A	27-10-1994
		WO 9424059 A	27-10-1994
		EP 0693041 A	24-01-1996
		ES 2111180 T	01-03-1998
		FI 954813 A	10-10-1995
		HU 73506 A	28-08-1996
		NO 954014 A	09-10-1995
		US 5720885 A	24-02-1998